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Title of the invention	Super plastic aluminium alloy production .
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Application date	26 May 1984
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Specification

1. Title of the invention

Super plastic aluminium alloy production

5 2. Scope of the patent claim

Super plastic aluminium alloy production where Al-Cu, Al-Mg, Al-Mg-Si or Al-Zn-Mg aluminium alloy ingot is thermally homogenized at from 400 to 550°C then thermally homogenized at from 300 to 550°C, and then, after it has
10 been hot-rolled at from 300 to 550°C, 1-stage or 2-stage heat retention is performed at from 350 to 550°C, and the system is then cooled at a rate of not less than 30°C/hr, and then, after at least 30% or more cold-rolling or from 20 to 60% cold-rolling has been performed, 300°C or cooler low-
15 temperature softening-annealing and cold-rolling are performed once or more; characterized in that a process is added whereby cross-rolling in a direction at right angles to the sheet is performed during the abovementioned hot-rolling and/or cold-rolling, such that when said cross-
20 rolling is performed during hot-rolling, rolling is performed to a rolling reduction of from 10 to 40% of the prescribed rolling reduction, and then rolling is performed to the remaining rolling reduction in a direction at right angles to said rolling, and when said cross-rolling is
25 performed during cold-rolling, rolling is performed, to a rolling reduction of from 10 to 40% of the prescribed rolling reduction for the cold-rolling process, in a direction at right angles to the abovementioned rolling direction.

30 3. Detailed description of the invention

[Field of industrial use]

The present invention relates to super plastic aluminium alloy production, more specifically, it relates to a method for producing super plastic aluminium alloy having
35 excellent super plastic extension.

[Prior art]

Super plasticity refers to the phenomenon whereby, under certain external conditions, material extends considerably, to from several hundred to several thousand percent, without necking, and this phenomenon can be broadly classified as fine grain super plasticity and transformation super plasticity, which involves constant temperature transformation. In order to reach super plasticity, the crystal size of the material must be kept fine. This can be achieved by altering the production conditions, for example, by optimising the alloy composition and altering the processing and heat treatment.

In the case of Al-Zn-Mg alloys, for example, material having an extension of 500% or more can be produced by taking Al-Zn-Mg alloy ingot comprising from 3 to 8 wt% of Zn and from 0.5 to 3 wt% of Mg as indispensable components and one or more chosen from not more than 3 wt% of Cu, from 0.05 to 2.0 wt% of Mn, from 0.05 to 0.5 wt% of Cr, from 0.05 to 0.5 wt% of Zr, from 0.05 to 0.5 wt% of V and not more than 0.15 wt% of Ti, with the remainder being Al and indispensable components, and thermally homogenizing said ingot at from 400 to 550°C then, after performing a heating process at from 300 to 500°C, performing a first heat retention at from 450 to 550°C for from 0.5 to 10 hr then cooling to a second heating temperature and performing a second heat retention at from 350 to 450°C for from 0.5 to 50 hr, then cooling at a rate of not less than 30°C/hr, and then, after at least 30% or more cold-rolling or from 20 to 60% cold-rolling, performing 300°C or cooler low-temperature softening-annealing and cold-rolling once or more, and then performing thermal softening-annealing at from 350 to 550°C at a rate of at least 100°C/hr.

This extension is limited in that, broadly speaking, (1) the super plasticity phenomenon is stopped by the coarsening of the crystal grain during reshaping and (2) voids (cavities) form due to stress concentration around the

crystallized compound and so the material breaks. The former situation can be improved by finely dispersing transition element intermetallic compounds such as $MnAl_6$, $Cr_2Mg_3Al_{18}$ and $ZrAl_3$, thereby inhibiting the coarsening of the crystal grain. As for the latter situation, firstly, the amount of crystals can be decreased by regulating the amount of Fe, Si and the like present, and moreover, the relationship between crystal size and stress concentration coefficient K is such that

$$K = f(a)\sqrt{\pi a} \quad (1)$$

a : external diameter of crystal

$f(a)$: shape factor dependent on the crystal size

and so, from equation (1), by decreasing the crystal size and by achieving even dispersion, it is possible to decrease the stress concentration around the crystals and to improve the super plasticity extension. Thus improvements in the properties of materials, in terms of their use in construction, can be anticipated.

[Problems to be overcome by the invention]

As described above, the present invention provides a super plastic aluminium alloy production method with which it is possible to further improve upon the super plasticity of conventional super plastic aluminium alloys.

[Means of overcoming the problem]

The special feature of the super plastic aluminium alloy of the present invention is, specifically, that

in super plastic aluminium alloy production where Al-Cu, Al-Mg, Al-Mg-Si or Al-Zn-Mg aluminium alloy ingot is thermally homogenized at from 400 to 550°C then thermally homogenized at from 300 to 550°C, and then, after it has been hot-rolled at from 300 to 550°C, 1-stage or 2-stage heat retention is performed at from 350 to 550°C, and the system is then cooled at a rate of not less than 30°C/hr, and then, after at least 30% or more cold-rolling or from 20 to 60% cold-rolling has been performed, 300°C or cooler low-

temperature softening-annealing and cold-rolling are performed once or more,

5 a process is added whereby cross-rolling in a direction at right angles to the sheet is performed during the abovementioned hot-rolling and/or cold-rolling, such that when said cross-rolling is performed during hot-rolling, rolling is performed to a rolling reduction of from 10 to 40% of the prescribed rolling reduction, and then rolling is performed to the remaining rolling reduction in a direction
10 at right angles to said rolling, and when said cross-rolling is performed during cold-rolling, rolling is performed, to a rolling reduction of from 10 to 40% of the prescribed rolling reduction for the cold-rolling process, in a direction at right angles to the abovementioned rolling
15 direction.

The super plastic aluminium alloy production of the present invention is described in detail below.

20 First, the heat treatment and processes in the super plastic aluminium alloy production of the present invention are described.

Ingot is obtained by casting Al-Cu, Al-Mg, Al-Mg-Si or Al-Zn-Mg aluminium alloy of prescribed composition and proportions according to common methods, then said ingot is thermally homogenized at from 400 to 550°C for long enough
25 to improve the hot-working properties and to homogenize the main elements that are non-uniformly distributed therein. Next, the material is hot-rolled at from 300 to 550°C to work the material to a prescribed sheet thickness, and some precipitates such as Zn, Mg and Cu and transition elements
30 such as Cr, V and Ti partially precipitate as the coarse casting composition becomes a hot-working fibre composition. If, during this hot-rolling, first rolling is performed to from 10 to 40% of the rolling reduction and then the slab is rotated and the remainder of the rolling reduction is
35 achieved by cross-rolling, the insoluble crystals comprising mainly Fe and Si and the insoluble crystals comprising

mainly Mn, Cr, Zr, V and the like are finely broken down, by the lengthwise and breadthwise cross-rolling, to a much greater extent than when cross-rolling is not performed, and the distribution becomes uniform. Stress forces around the crystals have a marked adverse effect on super plastic extension, and so as the abovementioned procedure alleviates said stress forces considerably, the resulting material exhibits greater extension. In the first rolling stage of said cross-rolling, the fine crystal formation and uniform dispersion effects are poor if the rolling is performed to less than 10%, whereas the crystal dispersion effects on rolling in the subsequent rolling direction are poor if the rolling exceeds 40%. Consequently, the rolling reduction for the first stage of the cross-rolling is from 10 to 40%.

After said hot-rolling, cold-working is performed, preferably to not less than 30%, which further increases the super plastic extension of the resulting fine grain material. Next, after said hot-rolling, heat retention is performed at from 350 to 550°C for from 0.5 to 20 hr, then cooling is performed at a rate of not less than 10°C/hr to force the solid solution elements to solid solution.

Said heat treatment may be performed at from 400 to 550°C for from 10 s to 10 min, using a continuous annealing furnace capable of rapid heating and rapid cooling. The Zn, Mg, Cu and the like become solid solution as a result of said heat retention, whereas the transition metal elements such as Mn, Cr and Zr precipitate as intermetallic compounds with Al, such as MnAl_6 , $\text{Cr}_2\text{Mg}_3\text{Al}_{13}$, ZrAl_3 , and the like. Super plasticity is not achieved and extension becomes difficult when the cooling after said 1-stage heat retention is performed at rates of less than 100°C/hr. When heat retention is performed in 2 stages, the first heat retention is performed at from 450 to 550°C for from 0.5 to 10 hr, then the system is cooled to the second heat retention temperature and the second heat retention is performed at from 350 to 450°C for from 0.5 to 50 hr, after which the

system is cooled at a rate of not less than 30°C/hr. Within said heat retention temperature ranges, the higher heat retention temperatures, and consequently the shorter times, are preferred.

5 Most of the soluble elements that were precipitated in the first heat retention form a solid solution in the second heat retention, then the transition elements such as Mn, Cr and Zr, and the intermetallic compounds with Al, such as MnAl_6 , $\text{Cr}_2\text{Mg}_3\text{Al}_{18}$, ZrAl_3 , precipitate as a result of the second
10 heat retention.

 When heat retention is performed twice, the transition element precipitation is fine and some high temperature precipitates (such as those comprising Al and Zn, Mg, Cu and the like) form, and so, even at the slow post-heat retention
15 cooling rate of 30°C/hr, production is easier and the dislocation density achieved during cold-working is higher than when heat retention is performed once.

 Super plastic extension becomes difficult if the cooling after said 2-stage heat retention is performed at
20 rates of less than 30°C/hr.

 These heat retentions are such that the composition at the lower part of the dislocation that results from the formation of hot-working fibre composition has its distortion energy decreased by recovery and
25 recrystallization, and dislocation is easily induced in the subsequent cold-working, and, as a result of the precipitated Mn, Cr and Zn particles, heating in the super plasticity temperature range after the subsequent cold-working allows the fine particle composition formed within
30 the material to be retained, and thus super plasticity is achieved.

 After cooling, cold-rolling is performed to at least 30% or more, but it is not possible to achieve a sufficiently fine crystal grain at less than 30% of the
35 working rate.

It is also possible to perform, once or more, the set of operations comprising a cold-rolling to from 20 to 60% followed by a low temperature softening-annealing at not more than 300°C, where the introduction of the low temperature annealing results in an even finer crystal grain. The cold-working cross-rolling to from 10 to 40% of the prescribed rolling reduction is performed during such cold-working because it breaks up the crystals, thereby improving the super plastic extension, and said cold-working cross-rolling is performed to from 10 to 40% for the same reasons as for hot-working cross-rolling.

Thus the composition of the lower part of the dislocation which has a high distortion energy is formed at a high density in the cold-worked material.

When this material is heated continuously, usually in a super plasticity temperature range at or above 0.5 T_m (where T_m is the melting point (absolute temperature) of the material) (400°C or higher for aluminium alloys), a new crystal grain forms as a starting point for the high density dislocation composition, and so the dislocation composition has a somewhat fine grain composition, becomes super plastic and its extension increases. Once recrystallization is complete, the crystal grain boundary energy decreases and so dislocation migrates and the crystal grain coarsens, and this coarsened composition adversely affects super plastic reshaping.

Accordingly, during thermal treatment in the super plastic aluminium alloy production of the present invention, dislocation migration is prevented and a fine grain composition is retained by controlling the dimensions and distribution of the precipitates such as intermetallic compounds, $MnAl_6$, $Cr_2Mg_3Al_{13}$ and $ZrAl_3$, which are formed as a result of the post hot-rolling heat retention. That is, the dislocation migration is not prevented if the precipitate dimensions are too small and/or the precipitate particle spacing is too large.

Moreover, material produced according to the super plastic aluminium alloy production of the present invention may be subjected to super plastic working in its cold-worked state, without further treatment, or after it has been cold-worked it may be heated at a rate of at least 100°C/hr, softened at from 350 to 550°C and subjected to super plasticity working.

The aluminium alloys which are the subject of the super plastic aluminium alloy production of the present invention are described below.

Al-Cu aluminium alloy comprises from 2 to 7 wt% of Cu as indispensable component and one or more chosen from not more than 2.5 wt% of Mg, not more than 2 wt% of Si, from 0.05 to 2.0 wt% of Mn, from 0.05 to 0.5 wt% of Cr, from 0.05 to 0.5 wt% of Zr, from 0.05 to 0.5 wt% of V and other than 0.15 wt% of Ti, with the remainder being Al and impurities.

Al-Mg aluminium alloy comprises from 2 to 7 wt% of Mg as indispensable component and one or more chosen from 0.05 to 1.5 wt% of Mn, from 0.05 to 0.5 wt% of Cr, from 0.05 to 0.5 wt% of V and not more than 0.15 wt% of Ti, with the remainder being Al and impurities.

Al-Mg-Si aluminium alloy comprises from 0.5 to 2.0 wt% of Mg and from 0.3 to 5.0 wt% of Si as indispensable components and one or more chosen from not more than 1 wt% of Cu, 0.5 to 1.5 wt% of Mn, from 0.05 to 0.5 wt% of Cr, from 0.05 to 0.5 wt% of Zr, from 0.05 to 0.5 wt% of V and not more than 0.15 wt% of Ti, with the remainder being Al and impurities.

Al-Zn-Mg aluminium alloy comprises from 3 to 8 wt% of Zn and from 0.5 to 3 wt% of Mg as indispensable components and one or more chosen from not more than 3 wt% of Cu, 0.05 to 2.0 wt% of Mn, from 0.05 to 0.5 wt% of Cr, from 0.05 to 0.5 wt% of Zr, from 0.05 to 0.5 wt% of V and not more than 0.15 wt% of Ti, with the remainder being Al and impurities.

It should be noted that if the amount of Fe or the amount of Si present as impurities exceeds 0.15 wt%,

insoluble crystals form and extension is decreased considerably, and so both the Fe content and the Si content are limited to 0.15 wt% or less.

[Working examples]

5 Working examples of the super plastic aluminium alloy production of the present invention are described below.

Working example

10 Ingots (400 mm thick) of the typical Al-Cu, Al-Mg, Al-Mg-Si and Al-Zn-Mg aluminium alloys shown in Table 1 were prepared by common DC casting, then sheet materials of a final thickness of 1.5 or 2.5 mm were produced by processing, and cross-rolling during hot-rolling and during cold-rolling, as shown in Table 2, and said materials were reshaped under the conditions described in Table 2.

15 As can be seen from Table 2, the extension due to super plastic reshaping was such that material produced according to the super plastic aluminium alloy of the present invention exhibited at least 80 to 15% better super plastic extension than material that had not been cross-rolled.

Table 1

No.	Alloy	Components (% by weight)										
		Si	Fe	Cu	Mn	Mg	Cr	Zn	V	Zr	Ti	Al
1	Al-Cu	0.06	0.10	4.5	0.6	1.5	-	-	-	-	0.01	R
2	Al-Mg	0.05	0.10	-	0.1	4.5	-	-	0.05	0.20	0.01	R
3	Al-Mg-Si	0.60	0.10	-	-	1.0	0.25	-	-	-	0.01	R
4	Al-Zn-Mg	0.05	0.10	1.5	-	2.3	0.20	5.7	-	-	0.02	R

R = remainder

Key to Table 2

	1	alloy
	2	ingot thickness (mm)
5	3	homogenization heat treatment ($^{\circ}\text{C} \times \text{hr}$)
	4	hot-rolling
	5	temperature ($^{\circ}\text{C}$)
	6	torque rolling reduction (mm)
	7	cross rolling reduction (%)
10	8	heating conditions ($^{\circ}\text{C} \times \text{hr}$)
	9	cooling rate ($^{\circ}\text{C/hr}$)
	10	final sheet thickness (mm)
	11	super plasticity properties
	12	distortion rate (1/s)
15	13	extension (%)
	14	none

[Advantages of the invention]

20 The super plastic aluminium alloy production of the present invention has the construction described in detail above and so it allows the production of material having excellent super plastic extension.

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Procedural Amendment (Voluntary)

5 June 1984

1. Indication of the case

Patent application number 107195 (1984)

2. Title of the invention

Super plastic aluminium alloy production

3. Party making the amendment

Relation to the case	Patent applicant
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5. Date of order to amend (voluntary)

26 June 1984 [stamp]

6. Subject of amendment

(1) The "Detailed description of the invention" section of the specification

7. Details of the amendment

(1) On page 12, line 10 of the specification, change "other than 0.15 wt% of Ti" to "not more than 0.15 wt% of Ti".

(2) On page 12, line 16 of the specification, change "from 0.05 to 0.5 wt% of V and not more than 0.15 wt% of Ti, " to "from 0.05 to 0.5 wt% of V, not more than 0.15 wt% of Ti and from 0.05 to 0.5 wt% of Zr, ".

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DERWENT-WEEK: 198605
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TITLE: Super-plastic aluminium alloy mfr. - comprises homogenising treatments of aluminium ingots hot rolling and cooling

PATENT-ASSIGNEE: KOBE STEEL LTD[KOBM]

PRIORITY-DATA: 1984JP-0107195 (May 26, 1984)

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INT-CL_(IPC): B21B003/00; C22F001/04

ABSTRACTED-PUB-NO: JP60251260A

BASIC-ABSTRACT: Mfr. comprises homogenising treatment of Al-Cu, Al-Mg, Al-Mg-Si, and Al-Zn-Mg system Al ingots at 400-550 deg.C, followed by 300-550 deg.C homogenising treatment; hot rolling at 300-550 deg.C, holding the heating temp. two or one steps in the range 350-550 deg.C, cooling with a rate of above 30 deg.C/hr., then cold rolling with over 30% or 20-60% draft, followed by low temp. annealing and cold rolling at least one time under 300 deg.C, where cross rolling is conducted on normal direction of the sheet material when hot rolling and/or cold rolling are made, e.g. in hot rolling, cross rolling is conducted in that manner, firstly rolled with 10-40% of required draft, then remaining draft is applied to the normal direction, i.e. toward that of the first rolling. While cold rolling, during the rolling process, 10-40% of the draft is applied in the normal direction toward rolling direction.

USE/ADVANTAGE - Al alloy has excellent super-plastic elongation The cross rollings are effective to give fine crystal grains in the structure, resulting in super-plasticity of the alloy (80-15% more elongation than that of non-cross rolled Al alloy).

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS:

SUPER PLASTIC ALUMINIUM ALLOY MANUFACTURE COMPRISE HOMOGENISE TREAT ALUMINIUM INGOT HOT ROLL COOLING

DERWENT-CLASS: M26 M29 P51

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審査請求 有 発明の数 1 (全7頁)

⑭ 発明の名称 超塑性アルミニウム合金の製造方法

⑯ 特 願 昭59-107195

⑰ 出 願 昭59(1984)5月26日

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明 細 書

1. 発明の名称

超塑性アルミニウム合金の製造方法

2. 特許請求の範囲

Al-Cu系、Al-Mg系、Al-Mg-Si系、
Al-Zn-Mg系のアルミニウム合金鋳塊を、
400～550℃の温度で均質化熱処理を行ない、
次いで、300～550℃の温度で均質化熱処理
を行ない、次いで、300～550℃の温度で熱
間圧延した後、350～550℃の温度において
1段階或いは2段階の加熱保持を行ない、30℃
/Hr以上の冷却速度で冷却してから少なくとも
30%以上の冷間圧延を行なうか或いは20～
60%の冷間圧延を行なった後に、300℃以下
の低温軟化焼鈍と冷間圧延とを1回以上行なう場
合において、前記熱間圧延時および/または冷間
圧延時に、板材に対し互いに直角方向のクロス圧
延を行ない、このクロス圧延を熱間圧延時には所
定圧下量の10～40%の圧下量で圧延を施した
後に、この圧延方向に対し直角方向に残りの圧下

量で圧延し、冷間圧延時には冷間圧延工程中に所
定圧下量の10～40%の圧下量で前記圧延方向
に対し直角方向に圧延する工程を付加することを
特徴とする超塑性アルミニウム合金の製造方法。

3. 発明の詳細な説明

〔産業上の利用分野〕

本発明は超塑性アルミニウム合金の製造方法に
関し、さらに詳しくは、超塑性伸びの優れた超塑
性アルミニウム合金の製造方法に関する。

〔従来技術〕

超塑性とは、ある外的条件の下で材料がくびれ
(necking)なしに、数百～数千%の巨大な伸びを
生じる現象であり、恒温変態を利用した変態超塑
性と微細粒超塑性に大別される。微細粒超塑性を
起させるためには、その材料の結晶粒径を微細に
制御することが必須である。これは、合金組成の
最適化と加工と熱処理等の製造条件により達成す
ることができる。

例えば、Al-Zn-Mg系合金では、Zn 3～8
wt%、Mg 0.5～3wt%を必須成分とし、Cu 3wt

%以下、Mn 0.05~2.0wt%、Cr 0.05~0.5wt%、Zr 0.05~0.5wt%、V 0.05~0.5wt%、Ti 0.15wt%以下の中から選んだ1種または2種以上を含有し、残部Alおよび不純物からなるAl-Zn-Mg系合金鋳塊を、400~550℃の温度において均質化熱処理を行ない、次いで、300~500℃の温度で熱間加工を行なった後、第1回の加熱保持を450~550℃の温度で0.5~10Hr行ない、次に、第2回の加熱温度まで冷却し、350~450℃の温度で0.5~50Hrの第2回の加熱保持を行ない、30℃/Hr以上の冷却速度で冷却してから、少なくとも30%以上の冷間加工を行なうか、或いは、20~60%の冷間加工を行ない、続いて300℃以下の低温軟化焼鈍と冷間加工を1回以上行ない、さらに、100℃/Hr以上の速度で350~550℃の温度で加熱軟化焼鈍を行なうことにより、伸びが500%以上である材料を製造することができる。

この伸びは大別すると、(1)変形中の結晶粒の粗大化による超塑性現象の停止および(2)晶出化

-3-

アルミニウム合金における超塑性をさらに向上させることが可能な超塑性アルミニウム合金の製造方法を提供するものである。

[問題点を解決するための手段]

本発明に係る超塑性アルミニウム合金の特徴とするところは、即ち、

Al-Cu系、Al-Mg系、Al-Mg-Si系、Al-Zn-Mg系のアルミニウム合金鋳塊を、400~550℃の温度で均質化熱処理を行ない、次いで、300~550℃の温度で均質化熱処理を行ない、次いで、300~550℃の温度で熱間圧延した後、350~550℃の温度において1段階或いは2段階の加熱保持を行ない、30℃Hr以上の冷却速度で冷却してから少なくとも30%以上の冷間圧延を行なうか或いは20~60%の冷間圧延を行なった後に、300℃以下の低温軟化焼鈍と冷間圧延とを1回以上行なう場合において、前記熱間圧延時および/または冷間圧延時に、板材に対し互いに直角方向のクロス圧延を行ない、このクロス圧延を熱間圧延時には所

合物の周りの応力集中によるポイド(キャビティ)の発生による材料の破断により制約を受ける。前者については、遷移元素の金属間化合物 $MnAl_6$ 、 $Cr_2Mg_3Al_{11}$ 、 $ZrAl_3$ 等を微細に分散させることにより、結晶粒の粗大化を抑制することにより改善でき、また、後者については、先ず、FeおよびSi等の含有量を規制することにより晶出物の量を低減することと、さらに、晶出物サイズと応力集中係数 K との間には、

$$K = f(a)\sqrt{\pi a} \quad \dots\dots (1)$$

a : 晶出物の外径

$f(a)$: 晶出物のサイズに依存する形状因子の関係があり、式(1)より晶出物のサイズを小さく、かつ、均一に分散させることにより、晶出物周りの応力集中を低減することが可能となり、超塑性伸びを向上させることができ、ひいては、構造材として使用していく上での材料特性の向上が期待できる。

[発明が解決しようとする問題点]

本発明は上記に説明したように、従来の超塑性

-4-

定圧下量の10~40%の圧下量で圧延を施した後、この圧延方向に対し直角方向に残りの圧下量で圧延し、冷間圧延時には冷間圧延工程中に所定圧下量の10~40%の圧下量で前記圧延方向に対し直角方向に圧延する工程を付加することにある。

本発明に係る超塑性アルミニウム合金の製造方法について以下詳細に説明する。

先ず、本発明に係る超塑性アルミニウム合金の製造方法における熱処理および加工について説明する。

Al-Cu系、Al-Mg系、Al-Mg-Si系、Al-Zn-Mg系の所定の含有成分および成分割合のアルミニウム合金を通常の方法により鋳造して得られた鋳塊は、内部に不均質に分布している主要元素の均質化および熱間加工性を向上させるため、400~550℃の温度において充分な時間均質化熱処理を行ない、続いて、300~550℃の温度で熱間圧延を行なって所定の板厚まで加工し、粗い鋳造組織は熱間ファイバー組織

となると同時に組織内にZn、Mg、Cu等の析出物およびCr、Zr、V、Ti等の遷移元素の一部が部分析出する。この熱間圧延時に、先ず、所定圧下量の10～40%の圧延を実施し、次いで、スラブを回転して残りの圧下量をクロス圧延で行なえば、FeおよびSiを主体とする不溶性の析出物およびMn、Cr、Zr、V等を主体とした不溶性の析出物は、縦横のクロス圧延を受けることにより、クロス圧延を実施しない場合よりも格段に微細に破壊され、その分布が均一となる。このようなことから、超塑性伸びを大きく阻害していた析出物周りの応力集中は大幅に緩和されて材料が大きい伸びを示すのである。このクロス圧延中、前段の圧延は10%未満では析出物の微細化、均一分散化に効果が少なく、また、40%を超える圧延では次の圧延方向における圧延による析出物の分散効果が少なくなる。よって、クロス圧延の前段圧下量は10～40%とする。

さらに、この熱間圧延後、好ましくは、30%以上の冷間加工を行なうことにより、さらに微細

-7-

2回の加熱保持において、第1回の加熱保持により析出している溶質元素はその大部分が固溶され、続く第2回の加熱保持により遷移元素のMn、Cr、Zr等とAlとの金属間化合物、 $MnAl_6$ 、 $Cr_2Mg_3Al_{11}$ 、 $ZrAl_3$ 等が析出する。

また、この2回の加熱保持は加熱保持を1回で行なった場合に比較して、遷移元素の析出形態が微細なことおよび若干のZn、Mg、Cu等とAlとの高温時効析出物が形成されるために、加熱保持後の冷却速度も30℃/Hrと遅くなくてもよく、製造がより容易となり、かつ、冷間加工中に生成される転位の密度がより高くなり、さらに微細な結晶粒が生成され超塑性伸びの大きいものが得られる。

この2段加熱保持後の冷却速度は30℃/Hr未満になると超塑性伸びが得られにくくなる。

これらの加熱保持により熱間ファイバー組織を形成していた転位の下部組織は回復、再結晶により歪エネルギーが低減され、続く冷間加工で転位が導入され易くなり、かつ、Mn、Cr、Zrの析

粒の材料が得られ超塑性伸びも大きくなる。次に、この熱間圧延後に350～550℃の温度で0.5～2.0Hrの加熱保持をしてから、100℃/Hr以上の冷却速度で冷却して固溶元素の強制固溶を図る。

また、この熱処理を急速冷却、急速加熱が可能な連続焼鈍炉により、400～550℃の温度で10sec～10min間行なってもよく、この加熱保持によりZn、Mg、Cu等は固溶され、一方、遷移元素のMn、Cr、Zr等はAlと金属間化合物の $MnAl_6$ 、 $Cr_2Mg_3Al_{11}$ 、 $ZrAl_3$ 等を析出する。この1段加熱保持後の冷却速度が100℃/Hr未満では超塑性が得られず伸びが出にくくなる。

また、加熱保持を2段で行なう場合、先ず、450～550℃の温度で0.5～1.0Hrの第1回の加熱保持を行ない、続いて第2回の加熱保持温度まで冷却し、350～450℃の温度で0.5～5.0Hrの第2回の加熱保持を行ない、30℃/Hr以上の冷却速度で冷却する。この加熱保持の温度が高い程時間は短時間でよい。

-8-

出粒子により、次の冷間加工後の超塑性温度域での加熱によって材料中に生成される微細粒組織が保持されて超塑性が得られる。

冷却後、少なくとも30%以上の冷間圧延を行なうのであるが、30%未満の加工率では充分微細な結晶粒が得られない。

また、20～60%の冷間圧延とこれに続く300℃以下の低温軟化焼鈍とを1回以上行なうこともでき、この低温焼鈍を導入することにより結晶粒はさらに微細化される。この場合の冷間圧延中に、所定冷間圧延量の10～40%の冷間クロス圧延を実施するのは析出物の破壊効果による超塑性伸び向上のためであり、この冷間クロス圧延量を10～40%とするのは、熱間クロス圧延の場合と同じ理由である。

このように、冷間加工された材料には、高い歪エネルギーを有する転位の下部組織が高密度に形成されている。

この材料を引き続き通常0.5Tm(Tmは材料の融点(絶対温度))以上の超塑性温度域(アルミニウ

ム合金では400℃以上)に加熱すると、高密度の転位組織を起点として新しい結晶粒が形成され、従って、転位組織は高密度である程微細粒組織が得られ超塑性となり伸びが大きくなる。しかして、一度再結晶が完了すると結晶粒界のエネルギーが減少するため転位が移動して結晶粒が粗大化し、そして、この粗大化した組織が超塑性変形を阻害することになる。

従って、本発明に係る超塑性アルミニウム合金の製造方法における熱処理においては、熱間圧延後の加熱保持により形成された金属間化合物、 $MnAl_3$ 、 $Cr_2Mg_3Al_{12}$ 、 $ZrAl_3$ 等の析出物の寸法と分布とを制御することにより転位の移動を阻止して微細粒組織を保持しているのである。即ち、析出物寸法が小さ過ぎたり、析出粒子間隔が大き過ぎると転位移動阻止効果が得られない。

また、本発明に係る超塑性アルミニウム合金の製造方法により製造された材料は、冷間加工したままの状態を超塑性の加工を行なってもよいが、冷間加工後、100℃/Hr以上の加熱速度で加

熱し、350～550℃の温度で軟化して超塑性加工を行なってもよい。

次に、本発明に係る超塑性アルミニウム合金の製造方法において対象となるアルミニウム合金について説明する。

Al-Cu系アルミニウム合金は、Cu 2～7wt%を必須成分として含有し、Mg 2.5wt%以下、Si 2wt%以下、Mn 0.05～2.0wt%、Cr 0.05～0.5wt%、Zr 0.05～0.5wt%、V 0.05～0.5wt%、Ti 0.15wt%以下の中から選んだ1種または2種以上を含有し、残部Alおよび不純物からなるアルミニウム合金である。

Al-Mg系アルミニウム合金は、Mg 2～7wt%を必須成分として含有し、Mn 0.05～1.5wt%、Cr 0.05～0.5wt%、V 0.05～0.5wt%、Ti 0.15wt%以下の中から選んだ1種または2種以上を含有し、残部Alおよび不純物からなるアルミニウム合金である。

Al-Mg-Si系アルミニウム合金は、Mg 0.5～2.0wt%、Si 0.3～5.0wt%を必須成分とし

-11-

て含有し、Cu 1wt%以下、Mn 0.5～1.5wt%、Cr 0.05～0.5wt%、Zr 0.05～0.5wt%、V 0.05～0.5wt%、Ti 0.15wt%以下の中から選んだ1種または2種以上を含有し、残部Alおよび不純物からなるアルミニウム合金である。

Al-Zn-Mg系アルミニウム合金は、Zn 3～8wt%、Mg 0.5～3wt%を必須成分として含有し、Cu 3wt%以下、Mn 0.05～2.0wt%、Cr 0.05～0.5wt%、Zr 0.05～0.5wt%、V 0.05～0.5wt%、Ti 0.15wt%以下の中から選んだ1種または2種以上を含有し、残部Alおよび不純物からなるアルミニウム合金である。

なお、不純物として含有されるFeおよびSi含有量は夫々0.15wt%を越えると不溶性の晶出物が生成し、伸びの低下が著しいのでFe、Si含有量は夫々0.15wt%以下に規制する。

[実施例]

本発明に係る超塑性アルミニウム合金の製造方法について実施例を説明する。

実施例

-12-

通常のDC鋳造法により第1表に示す代表的なAl-Cu系、Al-Mg系、Al-Mg-Si系、Al-Zn-Mg系のアルミニウム合金の鋳塊(厚さ400mm)を作製し、その後第2表に示す工程中、熱間圧延時、冷間圧延時にクロス圧延を実施して最終板厚1.5mmまたは2.5mmの材料を製造し、第2表に示す条件で変形した。

超塑性変形による伸びは第2表に示すように、本発明に係る超塑性アルミニウム合金の製造方法による材料が、クロス圧延を実施しない材料と比較して、超塑性伸びが80～15%以上向上することがわかる。

第 1 表

No	合金系	化 学 成 分 (重量%)									
		Si	Fe	Cu	Mn	Mg	Cr	Zn	V	Zr	Ti
1	Al-Cu	0.06	0.10	4.5	0.6	1.5	-	-	-	-	0.01
2	Al-Mg	0.05	0.10	-	0.1	4.5	-	-	0.05	0.20	0.01
3	Al-Mg-Si	0.60	0.10	-	-	1.0	0.25	-	-	-	0.01
4	Al-Zn-Mg	0.05	0.10	1.5	-	2.3	0.20	5.7	-	-	0.02

第 2 表

No	合金系	鋳塊厚さ (mm)	均質化 (℃×Hr)	熱間圧延			加熱条件 (℃×Hr)	冷却速度 (℃/Hr)	熱間圧延		最終板厚 (mm)	超塑性特性		
				温度 (℃)	トータル 圧延量 (%)	クロス圧 延量 (%)						温度 (℃)	ひずみ速 度 (1/sec)	伸び (%)
1	(2)Al-Cu	400	490×1240	420	400→6.3 (98%)	30	520×3+450×12	100	6.3→2.5 (60%)	なし	2.5	475	1×10 ⁻³	450
		"	"	"	"	なし	"	"	"	30	"	"	"	440
		"	"	"	"	なし	"	"	"	なし	"	"	"	340
2	(2)Al-Mg	400	510×1245	450	400→4.0 (99%)	30	540×3+420×10	100	4.0→1.5 (63%)	なし	1.5	550	2×10 ⁻³	590
		"	"	"	"	なし	"	"	"	30	"	"	"	565
		"	"	"	"	なし	"	"	"	なし	"	"	"	445
3	(2)Al-Mg-Si	400	465×1240	400	400→6.3 (98%)	30	510×3+400×10	100	6.3→2.5 (60%)	なし	2.5	510	2×10 ⁻³	440
		"	"	"	"	なし	"	"	"	30	"	"	"	415
		"	"	"	"	なし	"	"	"	なし	"	"	"	330
4	(2)Al-Mg-Zn	400	465×1240	400	400→6.3 (98%)	30	510×3+400×10	100	6.3→2.5 (60%)	なし	2.5	510	2×10 ⁻³	785
		"	"	"	"	なし	"	"	"	30	"	"	"	735
		"	"	"	"	なし	"	"	"	なし	"	"	"	580

【発明の効果】

以上詳細に説明したように、本発明に係る超塑性アルミニウム合金の製造方法は上記の構成を有しているものであるから、超塑性伸びが格段に優れた材料が製造できるという効果を有する。

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代理人 弁理士 丸 木 良 久

手続補正書（自発）

昭和59年06月25日

特許庁長官殿

1. 事件の表示

昭和59年特許願第107195号

2. 発明の名称

超塑性アルミニウム合金の製造方法

3. 補正をする者

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5. 補正命令の日付（自発）



-17-

6. 補正の対象

(1) 明細書の発明の詳細な説明の欄

7. 補正の内容

(1) 明細書第12頁10行の「Ti 0.15wt%

以化」を「Ti 0.15wt%以下」と補正する。

(2) 明細書第12頁16行の「0.15wt%以下

の中から」を「0.15wt%以下、Zr 0.05~

0.5wt%の中から」と補正する。